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**Structural Investigation of the Origin of Selectivity for Cs and Sr in the Nb-Substituted Titanosilicate with the Mineral Sitenakite Topology**

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Beamline(s): Highlight X7B

**Introduction:** The 25% niobium substituted crystalline silicotitanate with the composition  $\text{Na}_{1.5}\text{Nb}_{0.5}\text{Ti}_{1.5}\text{O}_3\text{SiO}_4 \cdot 2\text{H}_2\text{O}$  (Nb-CST) and having the mineral sitinakite topology shows significantly higher uptake value for  $^{137}\text{Cs}$  but lower for  $^{89}\text{Sr}$  than the CST [1]. To investigate the origin of selectivity, in-situ Cs and Sr ion-exchange studies were carried out.

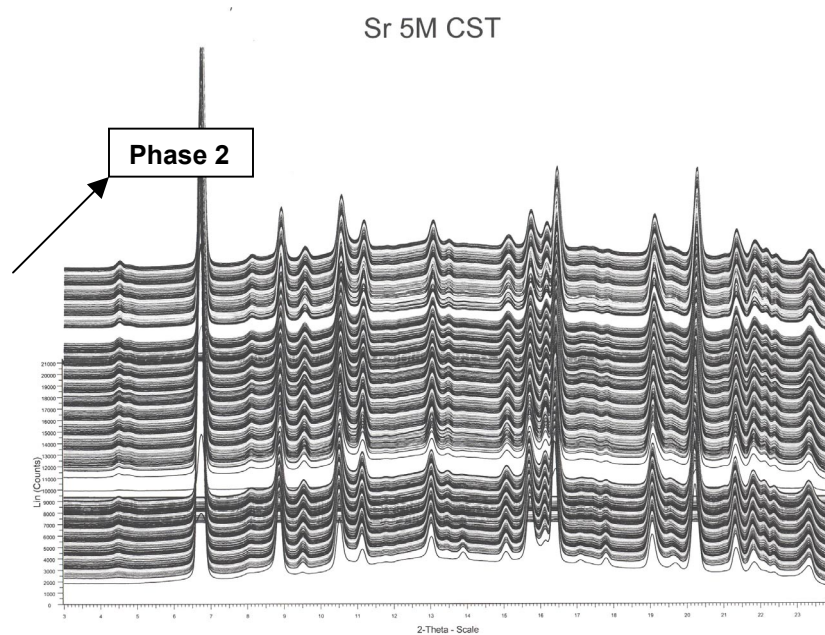
**Methods and Materials:** The ion-exchange reactions were carried out using a *Capillary-type cell* [2, 3]. The sample is locked between two glass wool plugs and the capillary is held in place using a Swagelock<sup>®</sup> fitting mounted on a goniometer head. The ion exchange on the H- and as synthesized-form of the titanosilicate,  $\text{Na}_{1.5}\text{Nb}_{0.5}\text{Ti}_{1.5}\text{O}_3\text{SiO}_4 \cdot 2\text{H}_2\text{O}$ , is performed *via* passing a desired concentrated solution into the capillary, controlled by the over pressure of  $\text{N}_2$  gas or vacuum applied to the other end of the capillary. During reaction, diffracted X-rays are collected on an online imaging plate detector.

**Results:** The changes in the observed powder diffraction pattern were analyzed by indexing them using the software NTREOR incorporated in the program EXPO [4]. The 25% Nb substitution, results in the increase in unit cell parameters and changes the population of water vs. Na in the channel to charge-balance the  $\text{Nb}^{5+} \leftrightarrow \text{Ti}^{4+}$  substitution, compared with the CST [5]. It appears that the coordination environment around Cs and Sr controls the selectivity of these radioisotopes. In-situ studies reveal two novel phases upon Sr exchange in CST. A slower exchange via less concentrated Sr solution and at a slower drop rate isolated these phases. Sr exchange in the Nb-CST showed no phase change. The Rietveld refinement of the model of Nb-CST indicated a seven coordinate Sr complex in the tunnel. Ex-situ ion-exchange is currently underway to isolate the two novel Sr phases obtained from the CST for structure solution.

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**References:**

- [1]. R. G. Anthony, R. G. Dosch, C.V. Philip, WO9419277 & US6110378.
- [2]. P. Norby, *Mater. Sci. Forum*, **147**, 228-231 (1996).
- [3]. C. L. Cahill, L. G. Benning, P. Norby, S. M. Clark, M. A. Schoonen, J. B. Parise, *Min. Mag.* **62A**, 267 (1998).
- [4]. A. Altomare, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, R. Rizzi & P. -E. Werner, *J. Appl. Cryst.* **33**, 1180-1186 (2000)
- [5]. D. M. Poojary, Roy A. Cahill, A. Clearfield *Chemistry of Materials*, **6**, 2364-2368 (1994)



**Figure 1.** Plot of the synchrotron X-ray powder diffraction profiles as a function of time during in situ Sr exchange of CST. The arrow shows the second phase that appears upon exchange.